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मानक

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IS 3520 (1992): Water-soluble sodium carboxymethyl cellulose [CHD 20: Paints, Varnishes and Related Products]



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सेलुलोज — विशिष्ट  
( दूसरा पुनरीक्षण )

*Indian Standard*

**WATER-SOLUBLE SODIUM CARBOXYMETHYL  
CELLULOSE — SPECIFICATION**

*( Second Revision )*

UDC 661.721.892.1 : 661.833

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**Price Group 5**

## FOREWORD

This standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Linters and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Sodium carboxymethyl cellulose, also known as cellulose gum and sodium cellulose glycolate, has a variety of uses in industries, such as paper, detergent, insecticides, textile and oil-well drilling. It is also used in food, medicine and cosmetics industries. Carboxymethyl cellulose is manufactured in several viscosity ranges and is also available in different degrees of substitution and active matter content depending on the end use. Taking this into consideration along with the prevalent trade practices and views of the users and manufacturers regarding the types in demand, it was decided to prescribe twelve types of water-soluble sodium carboxymethyl for the technical grade and six types for the pure grade cellulose. Each of the types has been given suitable designation for exact and easy identification by the purchaser. It is hoped that these types will cover all the present uses of this material satisfactorily.

This standard was first published in 1966. In the first revision in 1979, the pharmaceutical grade of the material, which was also suitable for cosmetic industry, was excluded from the standard.

In this second revision of the standard, alternative method for estimation of active matter content using uranyl nitrate has been provided. Identification testes for detecting water soluble gumes and starch have been added and Table 1 and Table 2 have been modified. In the technical grade, the active matter content has also been modified. Accordingly, the number of types for the technical grade has been reduced to twelve.

Determination of the degree of substitution by the method involving conversion of sodium salt of carboxymethyl cellulose of the acid form followed by its volumetric determination has been deleted. A new requirement for oil well drilling for insoluble matter in 6 percent sodium hydroxide solution has been incorporated and the limit for solubility in water has also been prescribed. Performance test for oil well drilling has been modified and Fann V G. viscometer has been recommended for determination of viscosity.

This standard does not cover the material used as thickening agent and stabilizer in food and pharmaceutical/cosmetic industry requirements for which are covered separately in IS 5306 : 1978 'Specification for sodium carboxymethyl cellulose, food grade (*first revision*)' and IS 9830 : 1988 'Specification for water soluble sodium carboxymethyl cellulose for cosmetic industry (*first revision*)'.

In the preparation of this standard, assistance has been derived from ASTM : D 1419-65 'Standard methods for testing sodium carboxymethyl cellulose', issued by the American Society for Testing and Materials, U. S. A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or a analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

# Indian Standard

## WATER-SOLUBLE SODIUM CARBOXYMETHYL CELLULOSE — SPECIFICATION

### ( Second Revision )

#### 1 SCOPE

This standard prescribes requirements and methods of sampling and test for water-soluble sodium carboxymethyl cellulose (CMC).

1.1 This standard covers the material used in textile, detergent, insecticide, oil-well drilling and paper industries.

#### 2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	Title
1070 : 1977	Water for general laboratory use ( <i>second revision</i> )
2316 : 1968	Method of preparation of standard solution for colorimetric and volumetric analysis ( <i>first revision</i> )
2317 : 1975	Method for gravimetric determination of sulphates ( <i>first revision</i> )
6186 : 1986	Specification for bentonite ( <i>second revision</i> )
12305 : 1988	Laboratory sintered (fritted) filters, porosity grading; classification and designation

#### 3 GRADES AND TYPES

3.1 The material shall be of the following two grades:

- a) Technical (T), and
- b) Pure (P).

3.2 The technical grade material having a degree of substitution either within the range of 0.4 to 0.55 or 0.55 to 1.0 and having a minimum active matter content of 55 percent shall be made available in six different viscosity ranges as extra low, low, medium, high, extra high and super high.

3.3 The pure grade material having a degree of substitution either within the range of 0.4 to 0.55 or 0.55 to 1.0 and having a minimum active matter content of 92 percent, shall be available in three different viscosity ranges low, medium and high.

3.4 Each type shall be given a designation indicative of the grade, range of viscosity, degree of substitution and percentage of active matter. The designation shall have two parts separated by a hyphen. The first part shall consist of code letters indicating grade and viscosity range and the second part shall consist of code number indicating degree of substitution and percent-

age of active matter. The basis of these designations is illustrated in Table 1.

3.4.1 There shall be altogether twelve types of technical grade and six types of pure grade of carboxymethyl cellulose.

#### 4 REQUIREMENTS

##### 4.1 Colour and Appearance

The material of both the grades shall be white to creamish powder. It shall be free from extraneous matter and other thixotropic materials, such as starches, gums or sodium alginate and shall be free-flowing, granular, fluffy or fibrous.

NOTE — The colour of the material for oil well drilling shall be as agreed to between the purchaser and the manufacturer.

##### 4.2 Moisture Content

The moisture content for both the grades shall be not more than 10 percent by mass at  $105 \pm 2^\circ\text{C}$  when determined according to the procedure prescribed in Annex A.

##### 4.3 Solubility in Water.

The material of both the grades shall be soluble in water to give a clear and colloidal solution and shall not have the matter insoluble more than 10 percent by mass when tested according to the method prescribed in Annex B.

##### 4.4 Active Matter Content

The active matter content shall not be less than 55 percent by mass for technical grade and not less than 92 percent for the pure grade. The active matter content shall be determined in accordance with procedure prescribed in Annex C.

##### 4.5 Degree of Substitution

The material of both the grades shall have the degree of substitution within the range of either 0.4 to 0.55 or 0.55 to 1.0 depending on the end use. The degree of substitution shall be determined by the method prescribed in Annex D.

##### 4.6 Detection of Starch and Water Soluble Gums

The material of both the grades shall be free from starch and water soluble gums when tested according to the method described in 4.6.1 and 4.6.2.

###### 4.6.1 Identification Test for Starch

Add two drops of 0.1 N iodine solution to 1 percent solution of carboxymethyl cellulose in water. Heat to

Table 1 Designations of Carboxymethyl Cellulose Types  
(Clause 3.4)

Grade		Viscosity		Degree of Substitution		Active Matter Content, Percent Min		Designation
Full Form (1)	Code (2)	Full Form (3)	Code (4)	Full Form (5)	Code (6)	Full Form (7)	Code (8)	(9)
Technical	T	Extra Low	EL	0.4 to 0.55	4	55	55	TEL-455
				0.55 to 1.0	8	55	55	TEL-855
Technical	T	Low	L	0.4 to 0.55	4	55	55	TL-455
				0.55 to 1.0	8	55	55	TL-855
Technical	T	Medium	M	0.4 to 0.55	4	55	55	TM-455
				0.55 to 1.0	8	55	55	TM-855
Technical	T	High	H	0.4 to 0.55	4	55	55	TH-455
				0.55 to 1.0	8	55	55	TH-855
Technical	T	Extra High	EH	0.4 to 0.55	4	55	55	TEH-455
				0.55 to 1.0	8	55	55	
Technical	T	Super High	SH	0.4 to 0.55	4	55	55	TSH-455
				0.55 to 1.0	8	55	55	TSH-855
Pure	P	Low	L	0.4 to 0.55	4	92	92	PL-492
				0.55 to 1.0	8	92	92	PL-892
Pure	P	Medium	M	0.4 to 0.55	4	92	92	PM-492
				0.55 to 1.0	8	92	92	PM-892
Pure	P	High	H	0.4 to 0.55	4	92	92	PH-492
				0.55 to 1.0	8	92	92	PH-892

NOTE — Last two degits shall indicate the percentage of actual active matter content in the designation.

boiling and keep at room temperature for about 30 minutes. Absence of blue colour shall indicate the absence of starch.

4.6.2 Identification Test for Water Soluble Gums

Determine the active matter content in carboxymethyl cellulose both by the gravimetric method and by the uranyl nitrate method as prescribed in Annex C. The difference in the active matter content by these two methods shall indicate the presence of other water soluble gums (guar-gum and other water soluble and alcohol insoluble gums).

4.7 Viscosity

The viscosity in centipoises of a two percent (m/v) solution of technical grade material and of one percent (m/v) solution of pure grade material shall be within the ranges indicated against each type in Table 2.

Table 2 Viscosity Ranges

Type Technical	Viscosity cP	Type Pure	Viscosity cP
Extra Low	0 to 29	Low	0 to 99
Low	30 to 99	Medium	100 to 599
Medium	100 to 249	High	Above 600
High	250 to 599		
Extra High	600 to 999		
Super High	Above 1 000		

4.7.1 The viscosity shall be determined by the method prescribed in Annex E using Brookfield Viscometer Type LVF.

NOTE — The actual range of viscosity shall be as agreed to between the purchaser and the manufacturer.

4.8 Additional Requirement for Oilwell Drilling

4.8.1 Viscosity

When the material is required for oil well drilling, it shall have viscosity of 30 cP, maximum, of a two percent solution (2 g of carboxymethyl cellulose, on dry basis, in 100 ml distilled water) at 30 ± 2°C, when determined by the method prescribed in Annex E using Fann V.G. viscometer.

4.8.2 Performance Test

The material when required for oilwell drilling shall pass the performance test as prescribed in Annex F.

4.8.3 Matter Insoluble in 6 percent Sodium Hydroxide Solution

The matter insoluble in 6 percent sodium hydroxide solution in carboxymethyl cellulose shall not be more than 10 percent on dry basis when determined by the method prescribed in Annex G.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in moisture-proof bags or

rigid containers as agreed to between the purchaser and the manufacturer.

## 5.2 Marking

Each bag/container shall be securely closed and shall bear legibly and indelibly the following information:

- Name of the material;
- Grade, type and designation of the material;
- Actual viscosity range and type of viscometer used;
- Indication of the source of manufacturer;
- Net mass;
- Batch number; and
- Month and year of manufacturing.

5.2.1 The containers may also be marked with the Standard Mark of the Bureau.

## 6 SAMPLING

The representative sample shall be drawn as per the procedure prescribed in Annex H.

## 7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

# ANNEX A

(Clause 4.2)

## DETERMINATION OF MOISTURE CONTENT

### A-1 PROCEDURE

Weigh about 5 g of the material in a tared dish to the nearest 0.1 mg. Spread the material uniformly over the bottom of the dish. Place the same in air oven maintained at  $105 \pm 2^\circ\text{C}$  for about two hours, cool in a desiccator and weigh. The sample is to be heated again for 30 minutes, cooled and reweighed. Heat the sample again for 30 minutes in an air oven, cool and reweigh. Continue till a variation in mass of not more than 5 mg for 30 minutes drying time is obtained. Preserve the material for determination of active matter content,

degree of substitution, matter insoluble, etc.

### A-2 CALCULATION

$$\text{Moisture content, percent by mass} = \frac{M - M_1}{M} \times 100$$

where

$M$  = mass in g of the material taken for the test, and

$M_1$  = mass in g of the material after drying.

# ANNEX B

(Clause 4.3)

## DETERMINATION OF WATER INSOLUBLE MATTER

### B-0 OUTLINE OF THE METHOD

A solution of the sample is prepared in the absence of carbon dioxide free water and centrifuged. The sediment is weighed as water insoluble matter.

### B-1 PROCEDURE

Weigh accurately an amount of sample equivalent to 2 g of the dry material in a beaker. Place a mechanical stirrer about 2.5 cm above the powder and add 160 ml of carbon dioxide-free water or boiled and cooled distilled water and stir for 30 minutes avoiding undue aeration of the solution and preventing access of carbon dioxide. Dilute to 200 ml with carbon dioxide-free water and allow to stand for 16 hours. Centrifuge 100 ml of homogenous solution at 3 000 rpm for 15 minutes or until all the fibre has settled; decant the supernatant liquid, replace it with an equal volume of

water, shake, and again centrifuge. Repeat the procedure until all the gelatinous material has dispersed. Replace the supernatant liquid with an equal volume of 0.01 N hydrochloric acid, stir and centrifuge. Finally replace the acid with an equal volume of acetone and centrifuge. Filter through a tared sintered glass crucible of porosity (p 40) (see IS 2305 : 1988) wash the residue with acetone, dry for 2 hours at  $105^\circ\text{C}$ , cool and weigh till constant mass is obtained.

### B-2 CALCULATION

$$\text{Water insoluble matter, percent by mass} = \frac{M_1 \times 100}{M}$$

where

$M$  = mass in g of the sample taken, and

$M_1$  = mass in g of the residue.



ANNEX C  
(Clause 4.4)

DETERMINATION OF THE ACTIVE MATTER CONTENT

**C-0** Two methods have been prescribed for determination of the active matter content, namely, the gravimetric method and the uranyl nitrate method. However, in case of dispute the uranyl nitrate method shall be the referee method.

**C-1 GRAVIMETRIC METHOD**

**C-1.1 Reagents**

The sodium carboxymethyl cellulose is precipitated from an aqueous solution by the addition of 95 percent ethanol. This is weighed and expressed as percentage of active matter content in the sample.

**C-1.2 Procedure**

Weigh about one gram of the material prescribed in A-1 (on bone dry basis) to the nearest 0.1 mg and place in a 250-ml beaker. Add 10 ml of hydrochloric acid and stir occasionally with glass rod. Add 20 ml of water and neutralize with sodium hydroxide using phenolphthalein as the indicator. Add about 0.2 ml of sodium hydroxide in excess. Precipitate the sodium carboxymethyl cellulose by adding 160 ml of 95 percent alcohol under vigorous agitation. Add the alcohol first slowly until a fine precipitate is formed, then add the rest. Allow the stirrer to run for about 15 minutes or until the precipitate settles rapidly and the supernatant liquid is clear. If a clear liquid is not obtained, place the beaker on a steam-bath for some time till the liquid becomes clear. Transfer quantitatively to an accurately weighed dry sintered glass filter crucible of porosity (p 40) (see IS 12305 : 1988) and filter by suction. Wash the beaker and the precipitate with 95 percent alcohol till the liquid is free from chlorides and finally, displace the alcohol by ether. Draw air through the crucible for about five minutes before placing it in a drying oven at  $105 \pm 2^\circ\text{C}$ . Dry to constant mass (one to two hours) and weigh after cooling in a desiccator. Preserve the precipitate for test in D-1.2.

**NOTE** — If the material still contains sulphates even after washing, prepare about 5 g of carboxymethyl cellulose material by following the procedure prescribed under C-1.2. The weighed quantity of the dried material shall be ashed and the sulphate content determined gravimetrically following the procedure prescribed in IS 2317 : 1975. The equivalent sodium sulphate content (mass of the barium sulphate  $\times 0.609$ ) shall be subtracted from the final value.

**C-1.3 Calculation**

$$\text{Active matter content, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

$M_1$  = mass in g of the dry precipitate, and

$M_2$  = mass in g of the material taken for the test.

**C-2 URANYL NITRATE METHOD**

**C-2.1 Reagent**

**C-2.1.1 Uranyl Nitrate Reagent**

Dissolve 40 g of the uranyl nitrate hexahydrate in 800 ml of distilled water and dilute to 1 litre.

**C-2.2 Procedure**

**C-2.2.1** Weigh accurately about 0.40 to 0.50 g of the material preserved in A-1 and transfer to a dry 500-ml beaker and moisten the dry material with alcohol (95 percent methanol or absolute methanol) and add about 100 ml distilled water and stir with glass rod to make solution with heating to 50 to  $70^\circ\text{C}$ . By means of a pipette add drop by drop 25 ml of uranyl nitrate reagent. Remove the heat source and continue stirring for about 5 minutes. Allow the precipitate to settle for more than 30 minutes.

**C-2.2.2** Decant the supernatant liquor through a tared glass filtering crucible of porosity (p 40) (see IS 12305 : 1988) and wash the precipitate in the beaker with three 200 ml portions of water. In the case of high viscosity carboxymethyl cellulose, allow the precipitate to settle for 30 minutes during each washing with 200 ml of water in order to avoid choking problem during filtration. Check the liquid for chlorides and sulphates. If present, continue the washing till the liquid is free of chlorides and sulphates. Finally transfer the precipitate to the crucible with about 10 ml portions of alcohol. Wash the beaker with alcohol and see that all the precipitate is transferred to the crucible. Remove as much liquor as possible from the precipitate by vacuum filtering apparatus. Dry the crucible in an oven at  $125\text{--}130^\circ\text{C}$  to a constant mass (usually about 1 hour is required) and weigh the precipitate. Record as UCMC found.

**C-2.2.3** Transfer the precipitate to a tared silica or porcelain crucible by removing as much material as possible from the glass crucible. Reweigh the crucible and the contents, and record the weight of the transferred precipitate as UCMC used. Ignite in a muffle furnace at  $750^\circ\text{C}$  to  $800^\circ\text{C}$  to the green uranium oxide  $\text{U}_3\text{O}_8$ . (Usually about 20-30 minutes are required for the ignition). Transfer the crucible with residue to a desiccator and weigh it. Record the mass of the residue as  $\text{U}_3\text{O}_8$ .

**C-2.3 Calculations**

$$\text{Uranyl fraction (U.F.)} = \frac{\text{Mass of } \text{U}_3\text{O}_8 \times 0.961}{\text{UCMC used}}$$

$$\text{D.S.} = \frac{162 \times \text{U.F.}}{135} - 192 (\text{U.F.})$$

$$\text{NaCMC}/\text{UO}_2(\text{CMC})_2 = 162 + \frac{80 (\text{D.S.})}{162} + 192 (\text{D.S.})$$

$$\begin{aligned} \text{Active Matter} &= (\text{UCMC found} \times \frac{\text{NaCMC}}{\text{UO}_2(\text{CMC})} \times 100 \\ (\text{NaCMC}), & \text{ percent by mass} = \frac{\text{mass of the material}}{\text{mass of the material}} \end{aligned}$$

## ANNEX D

### (Clause 4.5)

#### DETERMINATION OF DEGREE OF SUBSTITUTION

##### D-1 OUTLINE OF THE METHOD

The active ingredient recovered from the material is incinerated to leave a precipitate of sodium carbonate which is determined acidimetrically. The amount of sodium carbonate, being directly related to the amount of sodium carboxymethyl groups in the material, is calculated in terms of number of such groups per anhydroglucose unit and expressed as the degree of substitution.

##### D-2 REAGENTS

**D-2.1 Standard Sulphuric Acid** — 0.1 N (see IS 2316 : 1968).

**D-2.2 Standard Silver Nitrate Solution** — 0.1 N (see IS 2316 : 1968).

**D-2.3 Methyl Red Indicator Solution**

##### D-3 PROCEDURE

Weigh about 0.5 g of precipitate preserved in B-1.2 to the nearest 0.1 mg in a platinum crucible, ash very gently over a small flame or in a furnace maintained at 600°C to 650°C for about 30 minutes. Dissolve the ash in water and titrate against standard sulphuric acid

0.1 N in a conical flask with methyl red as the indicator. When the first, not very sharp, end point is reached, heat to boiling so that the carbon dioxide is removed and titrate to a new sharp end point. To ascertain that the ash does not contain sodium chloride, add potassium chromate and titrate with silver nitrate. If more than 3 drops of silver nitrate are required, the sodium chloride content of the isolated residue shall be determined and subtracted from the original mass of the residue. Similarly, if the ash contains sodium sulphate it is to be determined separately and the sodium sulphate content shall also be subtracted from the original mass of the residue (See IS 2317 : 1975).

NOTE — The method shall not be used for samples containing phosphate.

##### D-4 CALCULATION

$$\text{Degree of substitution} = \frac{0.162 B}{1 - 0.08 B}$$

where

$B$  = 0.1  $b/C$ ,

$b$  = volume in ml of standard sulphuric acid of 0.1 N required, and

$C$  = mass in g of precipitate taken.

## ANNEX E

(Clauses 4.7.1 and 4.8.1)

#### DETERMINATION OF VISCOSITY

**E-1** Two methods have been prescribed for determination of viscosity using Brookfield Viscometer Type LVF and Fann V.G. Viscometer. When the material is required for oil well drilling, the viscosity shall be determined by using Fann V.G. Viscometer.

##### E-2 DETERMINATION OF VISCOSITY WITH BROOKFIELD VISCOMETER

###### E-2.0 Outline of the Method

**E-2.0.1** The resistance to movement of a spindle is measured and expressed in terms of viscosity in centipoises.

###### E-2.1 Apparatus

**E-2.2.1 Brookfield Viscometer Type LVF**

**E-2.2.2 Mechanical Stirrer** — made of glass or stainless steel.

**E-2.2.3 Constant Temperature Bath** — maintained at  $30 \pm 2^\circ\text{C}$ .

###### E-2.3 Procedure

**E-2.3.1** Determine the moisture content of the material. Calculate on dry basis the mass of sample necessary to make 500 g of the test solution as follow:

$$\text{Mass in g of the sample} = \frac{100 \times A}{100 - B}$$

where

$A$  = desired dry mass in g of the sample, and

$B$  = percentage of moisture in the sample as weighed.

**E-2.3.2** Add 100 ml of water to the jar, add the quantity of sodium carboxymethyl cellulose as calculated under E-2.3.1 and then add sufficient water to make a total of 500 g of solution (2 percent solution m/m). Place the stirrer in the solution so that the blade is about halfway between the bottom of the jar and the surface of the liquid, stir till the sample dissolves. Remove the agitator and transfer the sample container

to the constant temperature bath and keep it there for 3 hours. Remove the sample container from the bath, stir it again and measure the viscosity with the Brookfield viscometer at  $30 \pm 2^{\circ}\text{C}$  selecting the proper spindle and speed from Table 3. Allow the spindle to rotate until constant reading is obtained.

**Table 3 Viscometer Spindles Required for Given Speeds**  
(Clause E-2.3.2)

Viscosity Range	Spindle No.	Speed rev/min	Scale	Factor
(1)	(2)	(3)	(4)	(5)
10 to 100	1	60	100	1
100 to 200	1	30	100	2
200 to 1 000	2	30	100	10
1 000 to 4 000	3	30	100	40
4 000 to 10 000	4	30	100	200

**E-2.4 Calculation**

**E-2.4.1 Viscosity (Brookfield)**  
in centipoises = Reading  $\times$  Factor

where

Reading = the number obtained from the viscometer, and

Factor = the number given in Table 3 for the spindle and speed selected.

**E-3 VISCOSITY USING FANN V.G. VISCOMETER**

**E-3.0 Outline of the Method**

The method is based on the principle that the torque exerted by a liquid is directly proportional to its viscosity. The apparatus comprises two cylinders. Suspension is contained in the annular space between the two cylinders. The outer cylinder or rotor sleeve is connected to a motor and is driven at a constant rotational velocity. The rotation of the rotor sleeve in the suspension produces a torque on the inner cylinder or bob. A torsion spring restrains the movement. A dial attached

to the bob indicates displacement of the bob. The instrument have constantly been so adjusted that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 and 600 rev/min. The apparent viscosity in centipoises equals the 600 rev/min reading divided by 2.

**E-3.1 Apparatus**

**E-3.1.1 Fann V.G. Viscometer (Direct Indicating Torsion Viscometer)**

The details of the viscometer shall be as follows:

- a) Speed factor — 0.5 ( 600 rpm )
- b) Torsion spring No. 1 — Spring No. F1 of 386 dynes/cm/deg
- c) Rotor — factor combine — 1.000

**E-3.1.2 Constant Temperature Bath** — maintained at  $30 \pm 2^{\circ}\text{C}$ .

**E-3.2 Procedure**

**E-3.2.1** Place 1 litre beaker containing 500 ml of distilled water on a magnetic stirrer having glass stirrer in the beaker. The water in the beaker is stirred slowly and then 10 g of the material as received is added. The addition of the material is to be done slowly to avoid formation of lumps. Stirring is continued for one and a half hours so that there is no splash of liquid and no entrapment of air takes place. The suspension formed is allowed to attain a temperature of  $30 \pm 2^{\circ}\text{C}$  by keeping in constant temperature bath.

**E-3.2.2** The suspension after attaining the desired temperature is poured in the annular space between the two cylinders of the Fann V.G. Viscometer. The outer cylinder or rotor sleeve is driven to attain a rotation of 600 rpm. Wait for the dial reading to reach a steady value. Record the dial reading for 600 rpm.

**E-3.3 Calculation**

**E-3.3.1 Apparent viscosity, Dial reading at 600 rpm**  
centipoise (CP) =

2

## ANNEX F (Clause 4.8.2)

### PERFORMANCE TEST FOR MATERIAL REQUIRED FOR OIL-WELL DRILLING

#### F-1 OUTLINE OF THE METHOD

The filtration loss shall be determined of the base mud with or without 1 percent material by using nitrogen gas for applying the pressure to assess the performance test.

#### F-2 APPARATUS

##### F-2.1 Electric Stirrer

Hamilton bench mixer or alike with single corrugated impeller of diameter 25 to 30 mm, having a speed of 17 000 to 18 000 revolutions per minute.

##### F-2.2 Filter Press

It consists essentially of a cylindrical mud cell of 75 cm inside diameter and at least 60 cm high. This chamber is made of materials resistant to strongly alkaline solutions and is so fitted that it becomes possible to

admit into and bleed from the top conveniently a pressure medium. Arrangement is also such that it is possible to place a sheet of 9 cm filter paper in the bottom of the chamber just above a suitable support. The filtration area is 45.8 cm<sup>2</sup>. Below the support is a drain tube for discharging the filtrate into a graduated cylinder. Sealing is accomplished with gaskets. The entire assembly is supported by a stand. It is possible to apply pressure with any non-hazardous fluid medium, such as gas. Presses are equipped with pressure regulator and may be obtained with portable pressure cylinder, midget pressure cartridges, or any means for utilizing hydraulic pressure. To obtain correlative results, uniform thickness of 9 cm filter paper, Whatman No. 50 or equivalent, shall be used. Figure 1 gives the filter press and cell assembly.

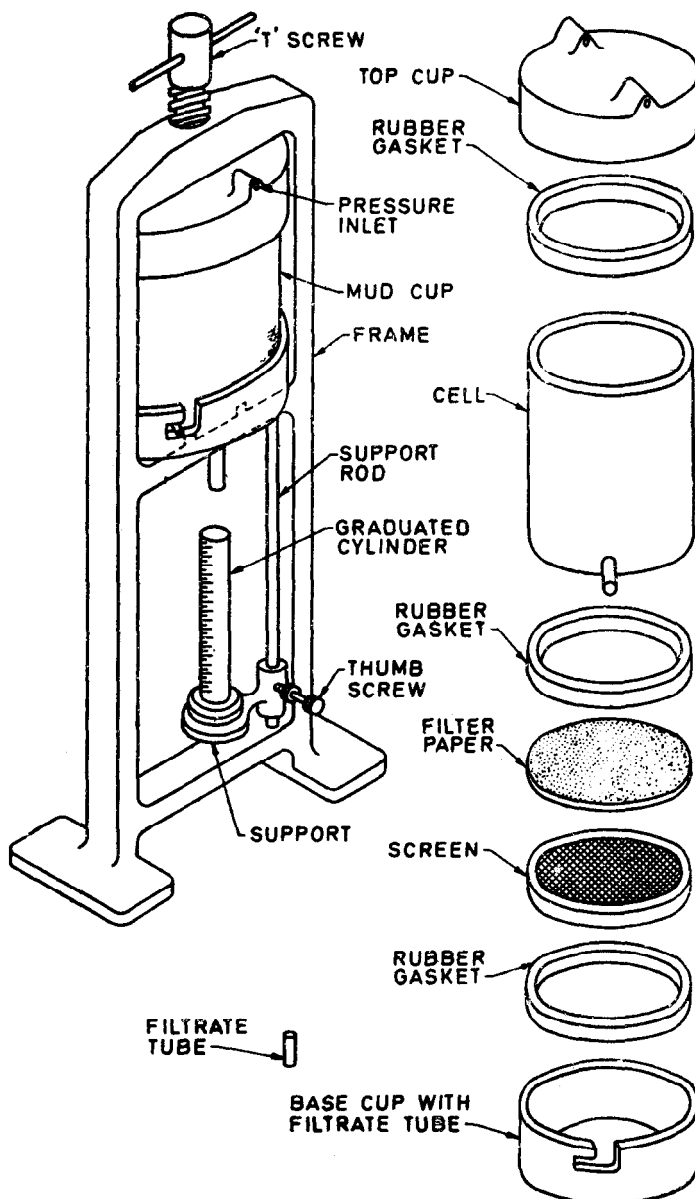


FIG. 1 STANDARD FILTER PRESS AND CELL ASSEMBLY

**F-3 REAGENTS****F-3.1 Salt Water**

Dissolve 40 g of sodium chloride (analytical grade) and 0.35 g of sodium bicarbonate (analytical grade) in 100 ml of distilled water.

**F-3.2 Bentonite** — Conforming to type 2 of IS 6186 : 1986.

**F-4 PROCEDURE**

**F-4.1** Mix 10 g of bentonite in salt water (see F-3.1) and made up to 100 ml with salt water by stirring in Hamilton Bench Mixture (17 000 to 18 000 revolutions per minutes in air) for 15 minutes. The base mud thus, obtained, is divided into two parts:

- a) One part of the base mud is aged at  $30 \pm 2^\circ\text{C}$  for 24 hours and the stirred for 20 minutes at 17 000-18 000 revolutions per minute in Hamilton Bench mixer for complete dispersion. Pour the dispersed material into the cell and complete the assembly. Place a dry graduated cylinder under the drain tube to receive the filtrate. Close the relief valve and adjust the regulator so that a pressure of 7 kgf/cm<sup>2</sup> (100 PSI) is applied in 30 seconds or less. The

test period begins at the time of pressure application. At the end of 30 minutes, measure the volume of filtrate. Shut off the flow through the pressure regulator and open the material and the relief valve carefully.

NOTE — The bentonite should be such that blank filtration loss is  $60 \pm 10$  cc and viscosity  $8 \pm 1$  cps in Fann V.G.

- b) To the remaining part of base mud 1 percent (1 g of carboxymethyl cellulose, as received, per 100 ml of the base mud suspension) is added and stirred at 17 000 to 18 000 rpm in Hamilton Beach mixer at  $30 \pm 2^\circ\text{C}$  for 20 minutes. The CMC treated mud is aged for 24 hrs at  $30 \pm 2^\circ\text{C}$  and then stirred at above speed in Hamilton Bench mixer at  $30 \pm 2^\circ\text{C}$  for 20 minutes. Follow the procedure as indicated in F-4.1(a) above.

**F-5 RESULT**

**F-5.1** The fluid loss value obtained in F-4.1(b) should not be more than 15 percent by volume of the value obtained under F-4.1(a) of the untreated Bentonite suspension. For instance, if the fluid loss in F-4.1(a) is 70 ml, then after treatment with CMC as described in F-4.1(b), the fluid loss should not be more than 10.5 ml.

## ANNEX G

(Clause 4.8.3)

### METHOD FOR DETERMINATION OF MATTER INSOLUBLE IN 6 PERCENT SODIUM HYDROXIDE SOLUTION

**G-1 REAGENTS**

**G-1.1 Sodium Hydroxide Solution** — 6 percent.

**G-1.2 Hydrochloric Acid** — 0.01 N.

**G-1.3 Acetone** — (See IS 170 : 1986).

**G-2 PROCEDURE**

Weigh accurately an amount of sample equivalent to 2 g of dry material in a beaker. Place a mechanical stirrer about 2.5 cm above the powder and add 200 ml of 6 percent NaOH solution. Keep this solution at 0 to  $4^\circ\text{C}$  for 2 to 3 hours, stir it again and keep for further 12 to 16 hours at ambient temperature. Centrifuge 100 ml of the solution at 3 000 rpm for 15 minutes or until all the fibre has settled; decant the supernatant liquid, replace it with an equal volume of 6 percent NaOH solution, stir and again centrifuge. Repeat the

procedure until all the gelatinous material has dispersed. Add 0.01 N hydrochloric acid to neutralize the suspension to 7 pH, stir and again centrifuge. Finally replace the supernatant liquid with an equal volume of acetone and centrifuge. Filter through a tared sintered glass crucible of porosity (p 40) (see IS 12305 : 1988), wash the residue with acetone, dry for about 2 hours at  $105 \pm 2^\circ\text{C}$ , cool and weigh till constant mass is obtained.

**G-3 CALCULATION**

$$\text{Carboxymethyl cellulose insoluble matter, percent by mass} = \frac{M_1 \times 100}{M}$$

where

$M$  = mass in g of the sample taken, and

$M_1$  = mass in g of the residue.

**ANNEX H***(Clause 6)***SAMPLING PROCEDURE FOR WATER-SOLUBLE SODIUM  
CARBOXYMETHYL CELLULOSE****H-1 LOT**

All the containers of the same grade and type of material in a single consignment belonging to the same batch of manufacture shall be grouped together to constitute a lot.

**H-2 METHOD OF SAMPLING**

**H-2.1** Each lot shall be sampled separately. The number of containers to be selected from a lot shall depend on the size of the lot and shall be in accordance with Table 4.

**Table 4 Number of Containers to be Selected  
from the Lot**

Number of Containers in the Lot			Number of Containers to be Selected
<i>N</i> (1)			<i>n</i> (2)
Up	to	5	All
6	to	50	5
51	to	100	8
101	to	300	13
301	to	500	20
501	to	1 000	32
1001	and above		50

**H-2.1.1** These containers shall be selected at random from the lot and in order to ensure the randomness of selection, random sampling methods given in IS 4905 : 1968 may be followed.

**H-2.2** From each container selected according to H-2.1, take approximately equal quantity of material from different portions of the bag so as to form a composite sample weighing about 250 g which shall be divided into three test samples, one each for buyer, seller and reference. Each test sample shall be transferred to a glass sample container and sealed air-tight. Label the containers with date of sampling and other particulars of the material and the lot.

**H-3 NUMBER OF TESTS**

The test for all the requirements shall be carried out on the sample as obtained in H-2.2.

**H-4 CRITERION FOR CONFORMITY**

The lot shall be considered to be in conformity with the requirements of this specification if all the test results obtained in H-3 satisfy the corresponding requirements as specified in this specification.

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